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Journal of Nuclear Materials 271&272 (1999) 376–380

journal of nuclear materials

Microstructural evolution in vanadium irradiated during ion irradiation at constant and varying temperature

K. Ochiai [a], H. Watanabe [b,*], T. Muroga [c], N. Yoshida [b], H. Matsui [d]

[a] *Interdisciplinary Graduate School of Engineering Sciences, Kyushu University, Kasuga, Fukuoka 816-8580, Japan*
[b] *Research Institute for Applied Mechanics, Kyushu University, Kasuga, Fukuoka 816-8580, Japan*
[c] *National Institute for Fusion Science, 322-6 Oroshi, Toki, Gifu 509-5292, Japan*
[d] *Institute for Materials Research, Tohoku University, Miyagi, Sendai 980-8577, Japan*

Abstract

To understand the influence of stepwise change of irradiation temperature on microstructural evolution in pure vanadium, ion irradiations were performed at 473/873, 673/873 and 873/473 K. The defect cluster density was strongly affected by the pre-irradiation temperatures. Suppression of interstitial type loop formation was prominent by pre-irradiation at lower temperatures. These results were explained by the appearance of vacancy-rich conditions at the beginning of the higher temperature irradiation due to the reclustering of the vacancies formed in the lower temperatures.

1. Introduction

Vanadium base alloys are considered to be a candidate low-activation material for fusion reactor devices. In addition, they generally shown a good resistance for void swelling which is typical for bcc materials. Since wall temperature of fusion devices is expected to change widely depending on operation history, the irradiation response of the components may be different from that predicted by isothermal experiments [1]. Our recent studies on Fe–Cr–Ni austenitic stainless steels under fission neutron irradiation [2,3] and Ni ion irradiation [4] showed that, when variation of temperature crossed a characteristic borderline temperature of microstructure evolution (573–623 K for Fe–Cr–Ni alloys), the pre-irradiation at lower temperature is very efficient for suppression of interstitial loop formation. The results were explained by the vacancy rich condition, which appears temporarily at the beginning of the high-temperature irradiation. But the border temperature of microstructure evolution strongly depends on the point defect behavior of the material.

In the present study, heavy ion irradiations of pure vanadium were carried out by changing temperature stepwise manner from low to high-temperature to investigate the basic mechanisms of microstructural evolution.

2. Experimental procedures

The result of chemical analysis of pure vanadium is shown in Table 1. Disk specimens for electron microscopy were wrapped with pure zirconium as a getter of oxygen and annealed for 2 h at 1373 K. 3 MeV Cu^{3+} ion irradiations were carried out with Tandem accelerator of Kyushu University in the temperature range of 473–873 K. After the irradiations, the area of damage peak (at around 700 nm) was electro-polished by back-thinning method. The damage rate at the damage peak was about 1.7×10^{-4} dpa/s. The implanted copper concentration in this region was about 10^{-3} at.% at 0.1 dpa.

In addition to constant temperature, irradiation temperature was changed cyclically between two

* Corresponding author. Tel.: +81-92 583 7717; fax: +81-92 5837 690; e-mail: watanabe@riam.kyushu-u.ac.jp.

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Table 1
The result of chemical analysis of pure vanadium used in this study

	Fe (ppm)	Ti (ppm)	Al (ppm)	N_2 (ppm)	Cr (ppm)	Si (ppm)	Ni (ppm)	O_2 (ppm)	V (%)
Pure V	120	54	100	60	54	40	9	13	99.9

temperatures. The combinations of the temperatures and fluences of the temperature variation experiments were 473 K (0.25 dpa)/873 K (0.5 dpa), 673 K (0.25 dpa)/873 K (0.5 dpa) and 873 K (0.5 dpa)/473 K (0.25 dpa).

3. Results

3.1. The microstructural evolution under constant temperature irradiations

Fig. 1 shows the irradiation temperature dependence of microstructure at 0.75 dpa. In this figure, upper and lower photos show a bright field and dark field image of microstructure, respectively. Fig. 2 shows the corresponding void contrast image at the same irradiation conditions. Inside–outside contrast technique showed that, in the lower temperature range below 673 K, small vacancy type. The Burgers vector of the defects was not detected in this study. The density of interstitial type dislocation loops decreased with increasing irradiation temperature. As shown in Fig. 1, needle-like precipitates, which were oriented along ⟨1 0 0⟩ directions, were observed at 773 and 873 K. The EDS analysis of the precipitate showed a slight enrichment of carbon. But small vacancy type dislocation loops, which were formed in the lower temperature, were not observed in the higher temperatures. Void formation was detected in the temperature range where the needle-like precipitates were observed. The needle-like precipitates formed at 873 K were stable up to 10 dpa and growth of the voids was not prominent up to 10 dpa. With increasing dose level above 10 dpa, the precipitates were dissolved and higher void swelling was observed. Fig. 3 shows the irradiation temperature dependence of the measured number density of loops, needle-like precipitates and voids. Since this figure shows that the microstructure was drastically changing at around 700 K, varying temperature irradiation experiments were performed in the temperature range above and below 700 K.

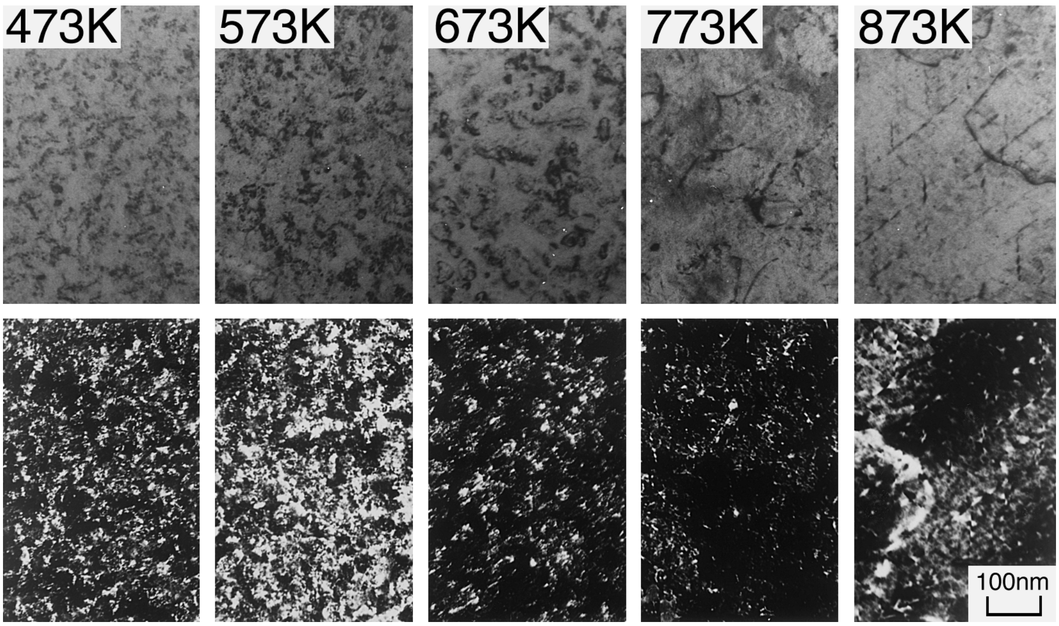

Fig. 1. Irradiation temperature dependence of microstructure at 0.75 dpa. Upper and lower photos show a bright field and dark field image of microstructure, respectively.

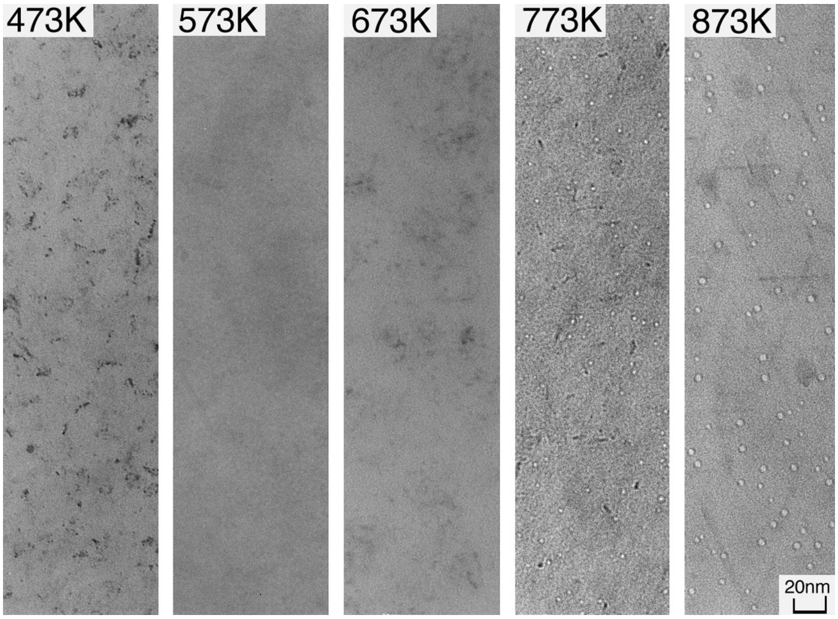

Fig. 2. Void contrast image of each irradiation temperatures at 0.75 dpa.

3.2. The microstructural evolution under varying temperature irradiations

In the case of stepwise change of irradiation temperature, the defect cluster density was strongly affected by the pre-irradiation temperature. Fig. 4 shows the microstructure of 473 K (0.25 dpa)/873 K (0.5 dpa) irradiation. The small loops formed at 473 and 673 K were dissociated by annealing and irradiation at 873 K. Especially in the case of pre-irradiation at 473 K, the loop density of 3×10^{22} m^{-3} at 473 K was decreased to 1×10^{20} m^{-3} by the irradiation at 873 K. The needle-like precipitates, which were observed at constant irradiation at 873 K, were also observed in the 873 K varying temperature irradiations. Fig. 5 shows the measured loop density before and after the irradiation at 873 K. Fig. 5(a) and (b) represent the case of 473 K (0.25 dpa)/873 K (0.5 dpa) and 673 K (0.25 dpa)/873 K (0.5 dpa), respectively. In the figure, the measured loop density after annealing at 873 K and the value at constant irradiation at 873 K (0.75 dpa) are also shown. From this figure, pre-irradiation at 473 K does not affect loop density at 873 K but by pre-irradiation at 673 K loop formation at 873 K was enhanced for these relatively low-dose studies.

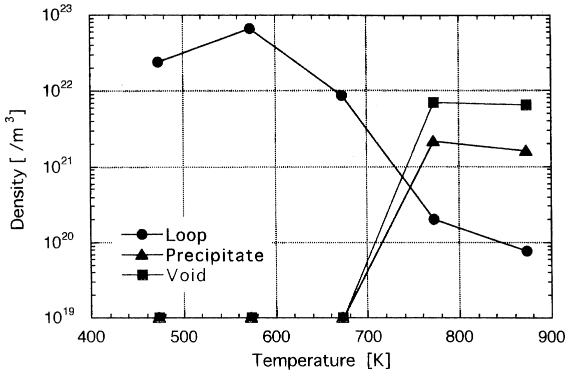

Fig. 3. Temperature dependence of measured defect density.

The precipitates formed at 873 K (0.5 dpa) were dissolved by irradiation at 473 K (0.25 dpa). The precipitate dissolution by the irradiation at 473 K is shown in Fig. 6 . By short irradiation at 473 K, precipitates formed at 873 K were dissociated and dense small loops appeared.

4. Discussion

In the constant temperature irradiations, voids were detected in the higher temperature range where the needle-like precipitates were observed. But growth of void was not prominent in the condition that these precipitates were observed. The needle-like precipitates formed at 873 K (0.75 dpa) were stable up to 10 dpa. These results indicate that the precipitates formed at higher temperature act as a strong unbiased sinks for point defects and thus are effective for void swelling resistance. The precipitates formed at 873 K (0.5 dpa)

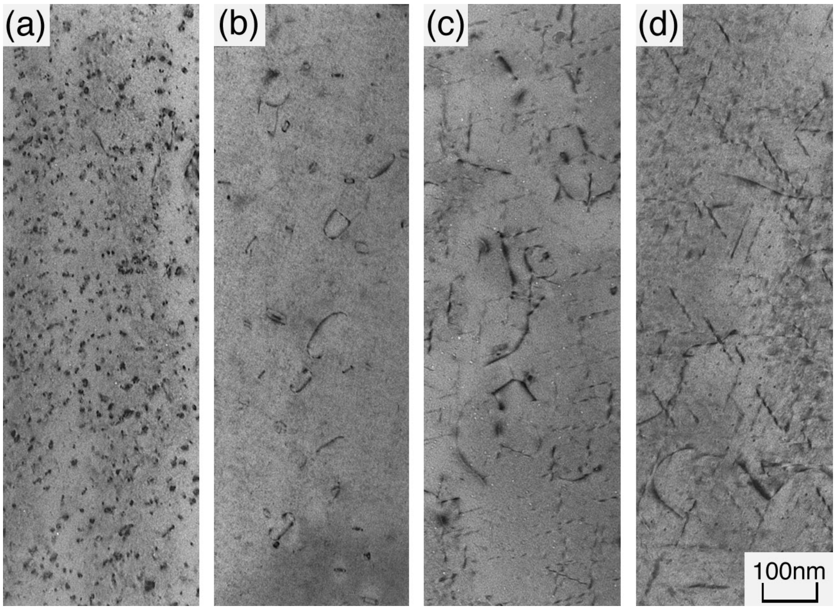

Fig. 4. Effects of temperature variation of 473 K (0.25 dpa)/873 K (0.5dpa): (a) 473 K, 0.25 dpa; (b) 473 K, 0.25 dpa + 873 K, 30 min anneal; (c) 473 K, 0.25 dpa + 873 K, 0.5 dpa; (d) 873 K, 0.75 dpa.

were dissolved by very low-dose irradiation at 473 K. This means that the void swelling resistance of pure vanadium might be affected by temperature variation from lower temperature and higher temperature due to the dissolution of fine precipitates.

As discussed in Refs. [2–5], the pre-irradiation at lower temperature is sufficient for suppression of interstitial loop formation in Fe–Cr–Ni austenitic stainless steels. The phenomena were explained qualitatively by the appearance of vacancy-rich condition at the beginning of the high-temperature irradiation due to the reclustering of vacancies formed by the low-temperature irradiation by reacting with radiation induced vacancies and interstitial. In this study, interstitial loops formed at lower temperature also disappeared by successive irradiation at 873 K. This result is also explained by the appearance of vacancy-rich condition at 873 K irradiation, which detected in Fe–Cr–Ni austenitic stainless steels.

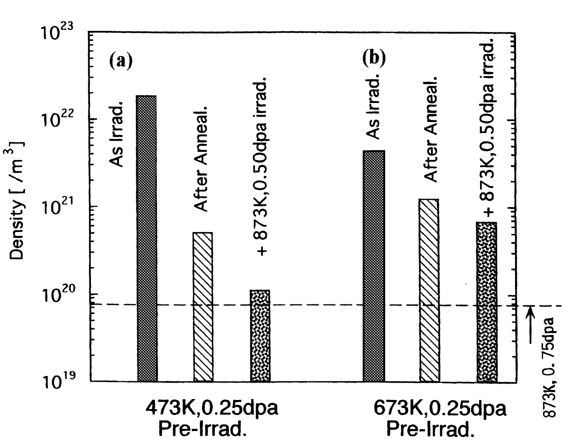

Fig. 5. Measured number density of interstitial type dislocation loops before and after the temperature variations: (a) 473 K (0.25 dpa)/873 K (0.5 dpa); (b) 673 K (0.25 dpa)/873 K (0.5dpa).

5. Conclusions

To understand the influence of irradiation temperature variation on microstructural evolution on pure vanadium, ion irradiation experiments were carried out in the temperature range of 473 and 873 K. Main results are summarized as follows.

1. The defect cluster density was strongly affected by the pre-irradiation temperatures. Suppression of interstitial type loop formation was prominent by pre-irradiation at lower temperatures.
2. The results were explained by the appearance of vacancy-rich conditions at the beginning of the higher temperature irradiation.
3. The needle-like precipitate formed at 873 K disappeared by successive irradiation at 473 K. Void swelling resistance of pure vanadium will be changed by the disappearance of these precipitates under temperature variation.

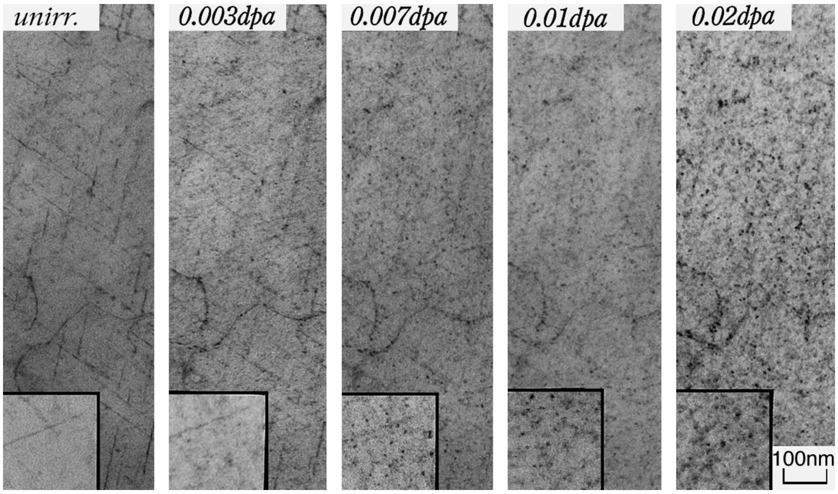

Fig. 6. Precipitate dissolution at 473 K.

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